

17p.

UNPUBLISHED PRELIMINARY DATA

N65 16977

(ACCESSION NUMBER)

17

(PAGES)

CR 52042

(NASA CR OR TMX OR AD NUMBER)

(THRU)

1

(CODE)

06

(CATEGORY)

2

(NASA CR 52042)

OTIS

FROM THE

(NASA Grant NSG-100-60)

GPO PRICE \$

OTS PRICE(S) \$

Hard copy (HC) 1.00

Microfiche (MF) .50

TITLE :

Interdisciplinary

Materials

Research

Center

PROGRAM

Five
Semi-annual
Progress Report,
1 Mar. - 31 Aug. 1963

RENSSELAER

POLYTECHNIC

INSTITUTE

TROY, N. Y.

7492002

25 Sep. 1963 17p

next p.

SPONSORED BY: National Aeronautics and Space Administration



[FIFTH] SEMI-ANNUAL PROGRESS REPORT [*] not p.

NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION

(NASA

GRANT

NSG-100-60

Rensselaer Polytechnic Institute
Troy, New York

auth (not p.)

September 25, 1963

Rensselaer Polytechnic Institute

~~F~~ FIFTH Semi-annual Progress Report
National Aeronautics and Space Administration Grant NsG-100-60
* March 1, 1963 to August 31, 1963

INTRODUCTION

This is the fifth semi-annual report from the Interdisciplinary Materials Research Program at Rensselaer Polytechnic Institute. During this report period one research project, "Diffraction Studies", was brought to a conclusion and one new program, "X-Ray Scattering" was started.

Construction of a new laboratory building to house the Interdisciplinary Materials Research Program will be started in the early fall. This building, which is being financed by a 1.5 million dollar grant from NASA, will provide adequate, optimal research space for the program which has far outgrown its present quarters in North Hall, and is now spread to other buildings through-out campus.

RESEARCH PROJECTS

Polymer Research

470.05

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A. Thermal Properties

A series of measurements of thermal conductivity of polyethylene-wax blends have been completed. These data complement earlier tests on linear polyethylenes. The results support a theoretical analysis of the dependency of thermal conductivity of linear polymers on molecular weight.

Further testing of the theory will be made by measuring thermal conductivities of a specially prepared series of polystyrenes of different molecular weights. A special apparatus is being built which will permit thermal conductivity measurements on elastomers in tension.

B. Morphology

Thin sections of annealed Nylon 66 stained with Phosphotungstic Acid and Chlorosauric Acid have been studied. Particular attention is being focused on the internal structure of spherulites. The next set of observations will be made on drawn and undrawn fibers of Nylon 66, and the neck region in partially drawn fibers.

C. High-Pressure Infrared Spectroscopy

The infrared spectra of polyvinyl alcohol and nylon 66 have been studied at pressures to 30,000 atmospheres. The hydroxyl group stretching frequency in PVA shifts to lower frequency with pressure by about 35 cm^{-1} per 30,000 atmospheres. The spectra of nylon are currently being analyzed. The high pressure spectra of copolymers of polyvinyl alcohol and polyvinyl acetate are now being studied. Calculations are being performed to relate macroscopic deformations to molecular modes of deformation.

D. Mechanical Properties

Data have been obtained on the effects of various solvents on the failure characteristics of several glass-like polymers. A theoretical treatment of the fracture mechanics of viscoelastic media is being numerically solved, and fracture data on polystyrene, poly (methyl methacrylate), and polycarbonates will be used to test the theory. (This work is partially supported by a grant from the Institute of Paper Chemistry.)

Dynamic mechanical spectra of various polar polymers are being studied, and a theory of mechanical response for polymer systems which are energy, not entropy, controlled, is being formulated. Solutions for isotropic media are now being extended to include anisotropic polymers. A generalized treatment of stress relaxation has resulted from these studies. (This work is being supported in part, by a grant from the National Science Foundation.)

E. Heat Transfer in Fibrous Structures

A new apparatus is being built for studying heat transfer in fibrous structures when partially liquid filled. This equipment will be used in a study aimed at elucidating the mechanisms of simultaneous heat and mass transfer in these systems. A primary objective will be to delineate the factors promoting high heat transfer rates with small temperature gradients. This work is supported by the National Science Foundation.

A Study of the Interaction of Dislocation with
the Discrete Second-Phase Particles in
Dispersion-Strengthened Alloys

470.09

Senior Investigator:	G.S. Ansell, Ph.D. Associate Professor of Metallurgical Engineering
Research Staff:	R.S. Goodrich, Jr., M.S. Met.E. Research Fellow C.J. Barton, B.Met.E. Graduate Assistant H.S. Kim, B.S. Graduate Assistant

This program is a broadly-based study of various aspects of dispersion-strengthening, directed toward achieving a basic understanding of the role of dispersion-strengthening in the materials field.

The role of a dispersed phase in affecting the mechanical behavior of crystalline solids is being studied by means of Transmission Electron Microscopy. The fundamental nature of yielding, work hardening, recovery, and creep behavior of dispersion-strengthened alloy systems is currently under investigation. Samples of two-phase alloys, produced both by precipitation and by powder-metallurgical techniques, are formed into thin foils and observed by means of the electron microscope during deformation at both room and elevated temperatures. These observations are then correlated with theoretical models which have been proposed to account for the strengthening effect of a second phase in such systems.

Studies to date have led to a general understanding of the overall substructural buildup during both rolling and tensile deformation. Some observations of discrete dislocation-particle interactions have provided verification of several aspects of the theoretical models previously proposed to explain the yielding behavior of dispersion-strengthened systems.

Work on the recovery, recrystallization, and creep behavior combined with further studies of the work hardening characteristics of two-phase alloys are currently being explored.

The following areas are currently under study within the framework of this overall program:

A. Strength of Ferrous Martensite

During the course of this program, a theoretical model has been proposed to account for the observed strengthening behavior of ferrous martensite. Critical experiments are now being performed, both to act as checks on the model and to further our understanding of the structure and strength of martensite. These experiments are concerned primarily with the variation in effects of a varying quench rate on steels, especially the variations in strength, in response to tempering, and in the amount

of austenite retained in the quenched structures. Quenching rates as great as $-50,000^{\circ}\text{F}/\text{sec.}$ have been achieved. Subzero microhardness measurements, X-ray and electron diffraction measurements, magnetic saturation curve determinations, and transmission electron microscopy have been and are being used to study the quenched structures. Studies to date provide a substantiation of the principle features of the proposed theoretical model.

B. Fracture Behavior

The fracture behavior of dispersion-strengthened alloys is being investigated through application of electron microscopy to surface replicas. This method, permitting as it does the examination of a fracture surface at very high magnification while retaining a great depth of field, allows an assessment of the mode of propagation of the crack which is both qualitative and quantitative. For the sake of writing brevity, the method is generally called "Electron Fractography", or "Electron Micro-Fractography".

Both impact and fatigue fractures have been investigated, and it was found that a distributed discontinuous second phase frequently serves as a source from which slowly propagating discontinuous cleavage cracks originate. This fracture behavior may play a profound role in the future applications of metals having a ductile matrix, but which have been dispersion-strengthened. Our studies suggest that the character of the particle-matrix interface is paramount in determining the fracture mode of two-phase systems.

C. Transmission Electron Microscopy

Several additional aspects of dispersion strengthening are being studied by means of transmission electron microscopy. Of particular interest is the nature of the grain boundary-dispersed-phase interaction in such alloys. The recrystallization behavior of alloys containing a stable dispersed phase is being observed in situ.

Study in each of these areas continues, and is expected to continue for another year. .

Nucleation in Liquid Metals

470.10

Senior Investigator:	W.J. Childs, Ph.D., Professor of Metallurgical Engineering
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The determination of the mechanical energy required for nucleation of supercooled metals has been continued in an effort to obtain a more accurate value and a better understanding of the nucleation phenomenon. A heavy

silicone flux has been used to contain the liquid metal in a vibrated crucible. This procedure effectively prevents splashing of the metal surface so that the energy distribution within the liquid is more uniform. High speed movies have been used to demonstrate the effect of surface disturbances during vibration on increasing nucleation. Average energy values obtained with the silicone flux were higher and more consistent than with a more fluid, non-containing flux.

Energy requirements for nucleating supercooled tin was also investigated. It was found that the nucleation of supercooled tin required less vibrational energy than bismuth when tested using the same flux.

Nucleation catalysis in supercooled bismuth was investigated by measuring the characteristic supercooling of a bulk specimen of bismuth in the presence of a gross catalytic substrate. Copper, chromium, manganese, iron and platinum were found to catalyze the nucleation of bismuth in descending order.

Ultrasonic Research

470.11

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Measurements are in progress on the third order elastic constants of two alkali-halide materials, NaCl and KCl. The procedure is to follow the changes in the propagation velocities for longitudinal and both transverse waves in the $[110]$ and $[211]$ directions while compressional force is applied down the $[111]$ axis. In general such a brute force procedure would fail because of plastic deformation. However, for this particular orientation of the stress axis there is zero resolved shear stress on all the primary slip planes, so that stresses of 10^8 dynes/cm² are possible without appreciable yielding. Values for the 6 third order constants for NaCl have been obtained and are being refined and checked for internal consistency. The analysis developed to handle this problem has also been helpful to workers at Oak Ridge who have been using high level ultrasonics to generate waves of the second harmonic.

The standard measurement of the 13 elastic constants of anthracene, mentioned in our last report, are being repeated for greater accuracy with specimens that have been carefully annealed. The annealing reduces attenuation, increases number and quality of echoes and thereby enhances the precision for velocity determination. Similar measurements are being carried out on naphthalene (likewise furnished by the Brush Co.). This material has a lower melting point and is well annealed at room temperature.

Ultrasonic Pulse Interferometry

470.12

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During the past six months, our research has been directed towards obtaining a reliable pressure calibration in our particular experimental arrangement, using several metals with well-established transition pressures and elastic-constant information. The calculations required to interpret the data have been programmed, greatly reducing the time required to obtain final results.

An exploratory investigation of the optical transmission as a function of pressure of various salts and minerals has been initiated, using a small diamond-anvil press. Polymorphic transitions in AgI, AgNO₃, and CaCO₃ (calcite → aragonite) have been observed.

Excited Species

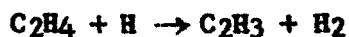
470.14

Senior Investigator: L.G. Bassett, Ph.D.
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Work has been in progress for studying the reactions of excited N₂ (A³Σ_u⁺) molecules. In the last six months it became obvious that a thorough investigation of the reactions of "active" nitrogen would be necessary as a precursor to any study of excited N₂ molecules. The following results and conclusions have been attained:

(1) we have proposed chain mechanisms for the reactions of C₂H₄, C₂H₂ and other hydrocarbons with N-atoms; these chains involve H-atoms and CN radicals such as the following:



(2) the concentration of N-atoms in active nitrogen as measured by NO titration, $\text{N} + \text{NO} \rightarrow \text{N}_2 + \text{O}$, gives values approximately twice that obtained from the amount of HCN formed when an excess of C_2H_4 is added to active nitrogen. We have proposed that this discrepancy is due to reactions of the CN radical in which N-atoms react to give molecular nitrogen. Some workers propose that excited N_2 molecules cause the discrepancy.

(3) the concentration of excited nitrogen molecules in active nitrogen is usually measured by the amount of ammonia decomposed since N-atoms do not react with NH_3 . We have shown that a radical, almost certainly CH, reacts with NH_3 to give HCN and H_2 as follows:



NH_3 is decomposed only slightly by active nitrogen from a condensed discharge and not at all from a microwave discharge. It may be, therefore, that small amounts of grease can form CH radicals that decompose the NH_3 .

(4) All attempts to observe reactions with our unique jet of excited $\text{N}_2(\text{A}^3\Sigma_u^+)$ molecules with many different reactants have been negative. We therefore conclude $\text{N}_2(\text{A}^3\Sigma_u^+)$ molecules react very slowly or not at all with the substances investigated.

In brief summary, we propose that N-atoms are the major reactants in active nitrogen and that excited N_2 molecules play a minor, if any, role in the chemical reactions of active nitrogen.

Mechanical Properties of Polymers

470.15

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A. Rheology

The flow curves of three additional ethylene-maleic anhydride copolymers (molecular weights: 25,000, 100,000, 500,000) have been obtained. These flow curves have been taken at 10°, 15°, 19°, and 25°C over a concentration range of 8 to 25 percent by weight copolymer in aqueous medium and over a shear rate range of 0 to 17,000 sec⁻¹. The data from these curves are being used to obtain correlations between energies of activation for viscous flow, viscosity, concentration and molecular weight, and to investigate the suitability of Bueche's shear rate molecular weight determination method for calculating molecular weights of these copolymers.

At this time the results are not complete, but indicate that the energies of activation decrease with an increase in molecular weight while increasing with an increase in the concentration.

Future work includes finishing compilations of the 500,000 molecular weight sample, and an examination of the correlations mentioned. A 250,000 molecular weight sample will also be studied. Investigation of these polymers with other solvents may also be undertaken, as well as the possible investigation of normal stresses which some of these samples exhibit.

B. Nature of Water of Hydration in Crystals

Infrared absorption spectra of 19 anhydrous and hydrated inorganic acetates have been measured from 4000 to 250 cm⁻¹ at both room temperature and at liquid nitrogen temperature. The number of coordinated water molecules per molecule of acetate was determined by Proton Nuclear Magnetic Resonance. In all appropriate cases heavy water hydrates have been prepared to confirm the absorptions due to the coordinated water molecules. The absorption bands have been assigned and correlated with the structure of the molecules as determined previously by X-ray diffraction methods. It is possible to distinguish bands resulting from different types of bound water. The spectra of these compounds yields valuable information concerning the nature of the metal-ligand bond. A variety of bands appearing in the far infrared region are probably caused by librational water modes. Work for the future includes measuring the remaining spectra and correlating these with the previous data.

C. Study of Inorganic Ruthenium Complexes Containing Neutral Ligands

The nature of the bonding of the SO₂ to ruthenium in these complexes will be studied to determine whether the SO₂ is coordinated through sulfur or oxygen and whether the S-O bond lengths differ from those in SO₂ itself.

The complexes $[\text{Ru}^{\text{IV}}(\text{NH}_3)_4(\text{SO}_2)\text{Cl}]\text{Cl}$, $[\text{Ru}^{\text{IV}}(\text{NH}_3)_4(\text{SO}_2)_2\text{Br}]\text{Br}$, and $[\text{Ru}^{\text{IV}}(\text{NH}_3)_5(\text{SO}_2)]\text{Cl}_2$ have been prepared. A complete three dimensional X-ray structure analysis of the Chloro-Chloride is underway. The space group is either Pna2, or Pnam, the latter being favored. Currently the locations of the principle scattering centers, viz., Cl, SO_2 and Ru, are being determined by the Patterson method. A check will be made on the other complexes to see if they are isomorphous with the chloro-chloride.

Following the X-ray work, as many bands as possible will be assigned in the 2-40 μ infrared region. It is also planned to look for other metals in the platinum group that may form SO_2 complexes, using analogous preparative procedures.

High Temperature Alloys

470.18

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A. Selected Topics Concerned with High Temperature Deformation and Rupture of Metals and Alloys

Through the courtesy of the General Electric Company Research Laboratories, more equipment has been made available on a loan basis which essentially doubles the testing capacity (creep and stress rupture) of the High Temperature Laboratory. Other, specialized, equipment now under construction will further increase capabilities. Work accomplished during the interim period has shown a real need for strain measuring instrumentation of greater sensitivity; hence, several units have been equipped with linear transducers and recording equipment.

Work having to do with the effect of the allotropic transformation of unalloyed cobalt, and cobalt-base solid solution alloys, has been continued. Further results must be obtained before a complete interpretation can be made. Some findings verify Russian reports, on other alloy systems, which show that ductility is increased as a phase boundary is approached.

Several solid-solution cobalt base alloys have been investigated as to strength and ductility, and some have been selected for further exploitation by possibly incorporating intermetallic (non carbide) precipitation hardening as a further strengthening mechanism.

B. Selected Studies of Transient Creep.

The role of recovery, as related to the time laws of transient creep, have been further investigated with three face centered cubic metals of minimum 99.994 weight percent purity. The occurrence of logarithmic and/or parabolic time laws has been shown to be stress and temperature dependent. Activation energies and volumes have been determined, and work is being continued.

In many regards the transient creep response of several solid solution alloys has been shown to be rather similar to that of "pure" metals. However, over certain stress and temperature ranges metallurgical phenomena occur which result in yield point formation and Luders band motion. Work is being continued.

C. Selected Studies of the High Temperature Oxidation of Metals and Alloys

Oxidation studies have indicated several solid solution cobalt base alloy systems which are sufficiently attractive to justify further exploitation, for strengthening, by incorporating precipitation hardening alloy additions in the compositions (see A, above).

Several findings of others having to do with the oxidation characteristics of unalloyed cobalt have been verified, and have been further extended. The multi-layer formation of CoO has been verified, under certain conditions of temperature and oxygen pressure, but the diffusing species have not been determined as yet. Oxidation characteristics are influenced by proximity to phase boundaries (allotropic transformation) and Curie points. Work with cobalt and cobalt base alloys is being continued.

Low Temperature Physics

470.19

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The studies of superconducting lead by means of the attenuation of ultrasonic waves have yielded two preliminary results. The first of these relates to the anisotropy of the energy gap in the allowed electronic states which characterizes the superconducting state. It appears that this energy gap for the electrons on a band of the Fermi surface perpendicular to the [100] direction opens more rapidly than for electrons on bands

perpendicular to the $[110]$ and $[111]$ crystalline directions as the temperature is lowered below that of the transition to superconductivity. Further measurements are now in progress to determine the limiting energy gaps at very low temperatures for these three bands.

Secondly, a most unexpected dependence of the attenuation of the superconducting lead upon the amplitude of the applied ultrasonic pulse has been found. Tests now underway should definitely settle the question of whether this effect is a property of the specimen or somehow a result of the instrumentation. All indications to date favor a real specimen effect but it is too early to speculate on its origin.

The studies of ultrasonic attenuation in γ -irradiated alkali halides have concentrated recently upon KCl crystals in order to be able to compare the results with previous observations in NaCl. The differences are considerable and, it is hoped, will lead to a better understanding of the previously reported post-irradiation annealing effects in these crystals.

Ceramics Research

470.20

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A. Self-Consistent Fields Across the 180° - Wall in BaTiO_3

The computer programs for the ferroelectric arrays are essentially completed and are being compiled. These programs, which are based on the twin symmetry of Ti-O coupling across the 180° - wall, will be run in the next few weeks. Among the first computer experiments to be tried will be a check of the effect of changes in the polarizability of the ions in the perovskite structure. A related calculation which was run to determine dispersion relations and oxygen polarizability in the solid state (particularly carbonate structures) has been completed and has demonstrated the validity of the point dipole concept (used in the BaTiO_3 case) when strong overlap is present. A smaller program to determine running time vs accuracy in the dipole-arrays programs has been run.

B. Heat of Reaction for the Decomposition of Al_2SiO_5 (Kyanite) to Al_2O_3 (corundum, plus SiO_2 (Silica).

From P-T data on the decomposition of Al_2SiO_5 to Al_2O_3 plus liquid, thermodynamic calculations are being made on the heat of this reaction for which there are conflicting data. Results indicate a value less than 10 kcal./mole. This is in good agreement with crystal chemical treatments but not with heat of solution measurements. The calculation is complicated by the fact that the liquid in the experimental study of the decomposition at high pressures and temperatures is not pure SiO_2 . It is hoped that if high pressure equipment can be made available other constant composition planes in the Al_2O_3 - SiO_2 system can be studied in order to better understand the relationships among the aluminum silicates at high pressure and temperature.

C. Cement Properites and Crystal Morphology

Using the litharge-glycerine system as a model cement, a direct correlation between the habit of the cement forming crystals and mechanical properties has been demonstrated. Radiating bundles of fine acicular crystals are associated with higher strength cement. This study is essentially completed and will be submitted for publication shortly.

Charge-Transfer Complexes

470.21

Senior Investigator:

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Graduate Assistant

Work on the spectral and thermodynamic properties of the benzene and diethyl ether molecular charge-transfer complexes with I_2 in the gas phase continues. The ultraviolet absorption peak associated with the benzene complex is located at or below 2700 Å, which is at least 270 Å below that of the comparable liquid-phase species; further investigation of the 2630-2700 Å region, with the hope of locating the peak precisely, is being undertaken. No similar shift of the iodine band in the visible region is observed. Preliminary studies indicate that the heat of formation of the benzene gas-phase complex is approximately the same as that of the liquid-phase complex.

The aliphatic amines are known to form relatively strong complexes with I_2 in the liquid phase, and therefore a study has been undertaken of the properties of these systems in the gas phase. In all cases investigated so far (triethyl, diethyl, and n-butyl amines), an irreversible reaction occurs leading to total disappearance of the I_2 and a water soluble product. Similar results have also been obtained for the isopropyl ether- I_2 system.

Dispersion-Strengthened Materials

470.23

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G. Leverant, Research Fellow

This project is concerned with the elucidation of dispersion strengthening and similar strengthening mechanisms in alloys. The first phase of an investigation entitled "Strengthening Behavior of Internally Oxidized Silver-Magnesium Alloys" has been completed. Dr. Bosch's thesis covering this subject has been accepted. It is planned to publish this work as a paper.

The study of internally oxidized silver-magnesium alloy is being continued. The influence of the rate of internal oxidation of the alloys upon the stability of the magnesium oxygen pairs in the "quasi" solid solution is being investigated. An apparatus has been built for internal friction studies of the alloys. This apparatus may also be used for measuring creep properties.

The study of the new concept of strengthening is being extended to other alloy systems. It was found that internally oxidized alloys of columbium with 1 weight percent of zirconium are becoming progressively stronger with increasing amounts of oxygen up to 0.5 weight percent (2.5 atomic percent). This behavior parallels that of pure columbium with increasing amounts of oxygen in solid solution. However, in alloys of pure columbium with oxygen, the lattice parameter of the columbium increases with increasing oxygen content, while in internally oxidized columbium - 1% zirconium alloys, no such increase, but rather a decrease in lattice parameter is observed. Micrographic studies are being undertaken to determine in what form the oxygen is present in these internally oxidized alloys.

Corrosion Studies

470.25

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The purpose of this program is to determine the electrochemical factors which control metallic corrosion. Funds have been used to partially support studies of the corrosion and electrochemical behavior of the rare earth metals and metallic passivity. Preliminary corrosion tests and the electrochemical measurements of the rare earths have been completed. It has been possible to qualitatively correlate the electronic

structure and chemical behavior of these metals. The measurement of active-passive behavior by potentiostatic techniques has been investigated. The effects of experimental variables on measured polarization behavior have been determined.

Future studies in the above areas include oxidation studies of the rare earth metals in dry and moist air, and hydrogen overvoltage characteristics of passive surfaces.

Nuclear Magnetic Resonance Research

470.26

Senior Investigator: P.A. Casabella, Ph.D., Assistant Professor of Physics

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The nuclear magnetic resonances of sodium nuclei in a single crystal of Rochelle salt have been studied at 28°C. The resonances from nuclei at each of the four types of sodium sites in the lattice have been observed and identified. These studies confirm that all four sites are chemically equivalent in the non-ferroelectric state. Similar experiments are now being carried out at 0°C, at which temperature Rochelle salt is ferroelectric. From studies of the differences between the nuclear magnetic resonances at the two temperatures, it should be possible to determine what changes take place in the chemical bonds at the sodium positions.

X-Ray Scattering

470.28

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The principal research effort of the "Diffraction" project is directed towards an intensive study of the elastic properties and interaction of elastic and magnetic behavior in certain metals and alloys by X-ray and neutron diffraction techniques. The systems under study, or to be studied include: Fe-Ni, Fe-Pt, Fe-Pd, Ni, Cr among others. X-ray Debye temperatures are measured both as a function of temperature (through the curie or Neel point, if such exists) and of composition (in the case of

alloys). In addition, Debye temperatures obtained from specific heat measurements by calorimetric methods will be used to complement the above data. The diffuse X-ray scattering in these systems will be thoroughly studied, under various conditions of temperature and composition, in order to obtain detailed information about the elastic properties. It is anticipated that similar studies using neutron diffraction will be undertaken in cooperation with NASA-Lewis (this idea having been initiated at the recent meeting in Cleveland). Both the X-ray and neutron work will involve measurements at liquid Helium temperatures.

The real time-computer control instrumentation to be used in obtaining iso-diffuse scattering data is continuing; the results of this work has been reported in a series of three papers at national and international meetings.

Work on the structure and physical properties of the group IIIb metal-trihalides is being continued.